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THESIS

PARTICLE SIZE DETERMINATION
USING A LASER LIGHT TRANSMISSION TECHNIQUE

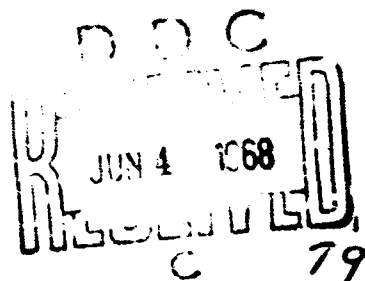
by

Jean Price Phelps

March 1968

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PARTICLE SIZE DETERMINATION
USING A LASER LIGHT TRANSMISSION TECHNIQUE

by

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ABSTRACT

A light transmission technique was used to measure the mean size of a group of graphite particles suspended in water and the particle sizes in an aerosol of ammonium chloride in air. Passage of a single red laser beam through a known concentration of graphite particles in water made it possible to obtain a measure of their mean size. Laser beams of two different wavelengths were used for the measurement of a suspension of unknown particle size and concentration.

Operating procedures were established for the apparatus, and calibration tests were performed in order to demonstrate the feasibility of the technique.

TABLE OF CONTENTS

SECTION	PAGE
I. Introduction	11
II. Light Transmission Theory	16
III. Light Transmission Experimental Apparatus	22
IV. Light Transmission Experimental Technique	28
V. Results and Discussion	32
VI. Conclusions	35
VII. Suggestions for Further Investigation	36
VIII. Bibliography	37
Appendix	
I. Procedures Checklist	61
II. Tables of the Mean Scattering Coefficient vs. Phase Shift Parameter for Several Values of m	63
III. Stokes' Law for Particle Size Determination	71

LIST OF TABLES

TABLE	PAGE
I. Sample Data Sheet	39
II. Graphite Particle Size Measurements	40
III. Data Sheet for the First Ammonium-Chloride Aerosol Experiment	41

LIST OF ILLUSTRATIONS

FIGURE		PAGE
1.	Experimental Arrangement Used by Durbin to Measure Particle Sizes in Ammonium-Chloride Fog by Light Scattering Methods	42
2.	Schematic Drawing of Optical Apparatus Used by Dobbins and Jizmagian for Light Transmission Measurements	43
3.	Optical Apparatus Used for Light Transmission Measurements of Solutions of Known Concentration but Unknown Size	44
4.	Optical Apparatus Used for Light Transmission Measurements of Solutions of Unknown Size and Concentration	45
5.	Spectral Response of the RCA 1P28 Photomultiplier Tube	46
6.	Dependence of Intensity on Concentration of Particles	47
7.	Graphite Particle Measurement - Run 12	48
8.	Graphite Particle Measurement - Run 13	49
9.	Graphite Particle Measurement - Run 14	50
10.	Graphite Particle Measurement - Run 15	51
11.	Graphite Particle Measurement - Run 16	52
12.	Transmission Measurement of Three Groups of Graphite Particles That Have Been Sorted by Size in a Settling Process	53
13.	Spectra Physics Model 124 Continuous Wave Laser with Diverging and Collimating Lenses	54
14.	TPW Pulsed Argon-Ion Laser with Diverging and Collimating Lenses	55
15.	Photomultiplier and Test Cell	56
16.	Instrumentation	57
17.	Equipment in Operation	58
18.	Test Cell Equipped to Generate Gaseous Suspensions	59

LIST OF SYMBOLS USED

T	Optical transmission
I	Intensity of light after it has passed through a group of particles
I ₀	Initial intensity of the light
λ	Wavelength of the light source
D	Particle diameter
α	Size number = $\frac{\pi D}{\lambda}$
m	Refractive index of the particles relative to the surrounding medium
ρ	Phase shift parameter = $2\alpha (m - 1)$
C _n	Number of particles per unit volume
C _v	Volume of particles per unit volume
l	Optical path length
K	Scattering coefficient
D ₃₂	Ratio of the third moment of the size distribution function to the second moment
ρ ₃₂	Phase shift parameter based on the diameter D ₃₂
RN	Reynolds Number
Note: Mean values are denoted by a bar (-) above the symbol	

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I. INTRODUCTION

With a steadily increasing interest in the subject of the atmospheric scattering of light and its parallel applications in certain fields of physics, meteorology, and colloidal chemistry, a sound knowledge of the scattering characteristics of particles is necessary.

A theory of light scattering by small particles has been derived from Maxwell's equations for the behavior of electromagnetic waves. The solutions to these equations in spherical coordinates were derived in 1908 by Gustav Mie. [18] This solution provides the basis for recent experiments in particle size determination using a light scattering technique. Experimentation by Penndorf [14], Kerker, et al. [9], and Meehan and Hugus [13], among others, has provided elaborate tabulation of the functions necessary for the solution of Mie's equation.

In contrast to most studies that have been concerned with dielectric spherical particles of constant diameter, the present investigation utilized particles of odd shapes and sizes. Hence, a mean diameter, as theorized by Dobbins and Jismagian [2] was the basis for particle size measurements in this study. Throughout this paper the term unknown suspension refers to a suspension whose chemical composition is known but whose particle sizes and concentration are unknown.

Particle Size Measurement Apparatus

There are two basic techniques in use today to determine particle sizes. The choice of technique depends solely upon the theory of operation—whether particle sizes are to be determined by measurement of scattered light or of transmitted light.

Schematic diagrams of experimental arrangements that have been used in measuring particle sizes are shown in Figures 1 - 4. Figure 1 is the arrangement used by Durbin [4] to determine particle sizes by measuring the intensity of light scattered by particles at different angles from the incident light. Figure 2 shows the arrangement used by Dobbins and Jismagian [2] to measure the transmission of light through a polydispersion of dielectric spheres. Dobbins and Jismagian have shown that light transmission measurements give more accurate results than those for light scattering measurements, especially where a study of the growth of particles is made.

Figures 3 and 4 show the arrangements used in this study. The principle of both is basically the same as that shown in Figure 2, with the following important exception. For this experiment lasers of known wavelengths were employed to eliminate the complexities of a monochromator and to insure a stable light source. The configuration shown in Figure 4 was constructed to enable the measurement of both particle sizes and concentrations in an unknown suspension. For this it was necessary to alternately measure the intensities of two lasers with one photomultiplier tube. Hence, a mirror and a beam-splitter were placed in the path of a second

laser beam in order to superimpose both beams on the photomultiplier tube.

In the numerous investigations of particle size measurement using light scattering techniques, all except a few have assumed a uniform size and shape of particles in order to utilize Mie's exact solution of the wave equation. However, the theory used here was that developed by Dobbins and Jismagian which is based on the observation that a particular mean diameter is directly related to the mean scattering cross section for a variety of particle size distribution functions and is relatively independent of the shape of the distribution function, which is usually unknown. This has been graphically shown for twenty different distribution functions, including rectangular, parabolic, and upper limit. [2] The agreement between the various values of \bar{K} , as represented by the standard deviation between each of the curves of \bar{K}/ρ_{32} vs ρ_{32} , was poorest at ρ_{32} equal to about 4.0 at which the standard deviation is 5.6 per cent. For other values of ρ_{32} the standard deviation was much less, especially for values of $\rho_{32} > 10$ where the standard deviation is approximately equal to zero. Hence, for the suspension of graphite particles used in this experiment ($\rho_{32} \gg 10$), any of the distribution functions would provide accurate results. This line of reasoning was followed because it allowed a flexible application of the light transmission theory to nonuniform particle sizes.

Description of Experiment

The purpose of this project was twofold. One was to develop a facility for the determination of particle size and concentration

in the exhaust of a rocket being developed at the Naval Postgraduate School. The second was to continue the work of Dobbins and Jismagian by determining the relationship of the theorized mean diameter of a group of particles suspended in a medium to the physical characteristics of the particles.

In this thesis the results of two experiments are described. Graphite particles of known sizes, similar to those expected in the rocket exhaust, were immersed in water in discrete amounts to provide known concentrations. Also, an aerosol of ammonium-chloride particles was generated for which neither particle size nor concentration was known. Experiments were made on both groups of particles.

In the first experiment, measured quantities of graphite particles were added to water in an optical cell, and the transmission of a laser beam through the cell was recorded. The mean particle size was determined by employing the theory developed by Dobbins and Jismagian. In the second experiment, prior measurement of the concentration of the particles in an aerosol of ammonium-chloride was not possible. However, both the mean particle size and the concentration could be determined by measuring the light transmission of two lasers of widely separated wavelengths.

Completion of this project included the following:

- a. A light transmission system, including an associated measurement facility, was assembled.
- b. An optical cell, equally as suitable for liquid solutions as for gas dispersions, was designed and assembled.

c. The system was instrumented to insure that uniform, reproducible conditions existed for successive experiments.

d. Techniques were developed for sorting graphite dust samples into uniform sizes and for measuring their volume.

e. Sufficient tests were performed to reach a conclusion as to the applicability of this technique for measuring particle sizes in a rocket exhaust and for determining the relationship between the theorized mean diameter and the diameters observed under magnification.

f. Procedures for the operation of the system as a whole were established.

g. Mean scattering coefficients were determined for the relative refractive indexes of a solution of graphite and water ($m = 1.673$) and an aerosol of ammonium-chloride particles ($m = 1.642$).

II. LIGHT TRANSMISSION THEORY

When a parallel beam of light is incident upon a group of spherical particles of uniform size, a fraction of the incident light is extinguished by the particles through absorption and scattering. This scattering is the means by which the particles are visible. Since the light absorbed by a group of particles is so much less than the scattered light, the fraction of this parallel beam of light that emerges without experiencing a deflection from the initial direction of propagation is given by

$$T = \frac{I}{I_0} = \exp[-K(\alpha, m) \frac{\pi}{4} D^2 C_n l] \quad (1)$$

where

I/I_0 is the fraction of the light intensity which passes through the particles unchanged

T is the optical transmittance

$K(\alpha, m)$ is the scattering coefficient for the particles

α is the size number ($\alpha = \frac{\pi D}{\lambda}$)

λ is the wavelength of incident light

m is the refractive index of the particles relative to the surrounding medium

D is the particle diameter

C_n is the concentration or number of particles per unit volume

l is the optical path length.

Equation (1) can be used to test the solution developed by Gustav Mie in 1908 for Maxwell's wave equations for the condition of an electromagnetic wave incident on a sphere. Through experimentation, the scattering coefficient can be calculated for a group of spherical

particles of known concentration and diameter. This scattering coefficient has been compared with that given by the Mie Theory, with an agreement of 10 per cent or better for a broad range of $\bar{K}(\alpha, m)$. [2] Therefore, it can be assumed that use of the Mie Theory will yield valid results for the scattering coefficient of an unknown suspension.

In reality, however, few dispersions are of uniform size. Therefore, the transmittance law as given by Equation (1) has little real significance in practical applications. To make Equation (1) valid for a variety of distribution functions, it must be assumed that there exists a particular diameter which is directly related to a mean scattering cross section and which is independent of the shape of the size distribution function; see Equation (4). The transmission equation used for these experiments follows the development advanced by Dobbins and Jizmagian [2] and is partially reproduced here.

Equation (1) can be extended to a polydispersion by determining a scattering coefficient as a function of the particle diameter and some particle size distribution function $N_r(D)$ such that

$$\int_{D_2}^{D_1} N_r(D) dD \quad (2)$$

is the relative probability of the occurrence of sizes between D_1 and D_2 . Therefore, Equation (1) becomes

$$T = \exp \left[- \frac{\pi}{4} C_n \int_0^{D_m} K(D, m) N_r(D) D^2 dD \right] \quad (3)$$

where D_m is the maximum particle diameter present. A mean

scattering coefficient may then be defined as

$$\bar{K} = \frac{\int_0^{D_m} K(D,m) N_r(D) D^2 dD}{\int_0^{D_m} N_r(D) D^2 dD} \quad (4)$$

To eliminate the obvious complexities of accurately counting a very large number of small particles, the number concentration may be replaced by a volume concentration C_v , i.e., the volume of particles per unit volume, such that for spherical particles

$$C_v = C_n \frac{\pi}{6} \int_0^{D_m} N_r(D) D^3 dD \quad (5)$$

Inserting Equations (4) and (5) into Equation (2), the remaining integral quantity is the ratio of the third moment of the size distribution function to the second moment, or the volume to the surface mean diameter, such that

$$D_{32} = \frac{\int_0^{D_m} N_r(D) D^3 dD}{\int_0^{D_m} N_r(D) D^2 dD} \quad (6)$$

Introducing Equation (6), the transmittance laws can be rewritten as

$$T = \exp \left[- \frac{3}{2} \left(\bar{K} / D_{32} \right) C_v l \right] \quad (7)$$

A simple interpretation of D_{32} is obtained by noting that the volume-to-surface ratio of a collection of particles of uniform size is $D/6$. The volume-to-surface ratio of a collection of C_n particles per unit volume is

$$\frac{C_n \frac{\pi}{6} \int_0^{D_m} N_r(D) D^3 dD}{C_n \int_0^{D_m} N_r(D) D^2 dD} = \frac{D_{32}}{6} \quad (8)$$

Thus the volume-surface mean diameter is that diameter which is exactly six times the volume-to-surface ratio of the polydispersion. [2]

Equation (7) and the volume concentration, therefore, would be sufficient to determine the quantity \bar{K}/D_{32} and thus provide a measure of some mean particle size. Evaluating Equation (7) for two widely separated waves of light through unknown suspension would produce mean particle diameter and concentration.

Evaluation of \bar{K} is a complete investigation in itself and will only be touched upon lightly. Let it suffice to say that the mean scattering coefficient which is normally given by a slowly converging series can be approximated by

$$K = 2 - (4 \sin \rho/\rho) + 4(1 - \cos \rho)/\rho^2 \quad (9)$$

for $m - 1 \ll 1$ and $\alpha \gg 1$, where $\rho = 2\alpha (m - 1)$ is the phase-shift parameter. Thus for particles much larger than the wavelength of the incident light

$$\frac{\bar{K}}{\rho_{32}} \rightarrow 2/\rho_{32} \quad (10)$$

$\alpha \gg 1$

Most of the graphite particles used for the first experiment were found to be much larger than the wavelength of the lasers used. Therefore, \bar{K} was set equal to 2. Where this simplifying assumption could not be made, values of \bar{K} vs ρ_{32} were extracted from tables interpolated from those provided by Dobbins and Jismagian. [2] The original values were tabulated by using Equation (4) and the upper limit function

$$N_T(D) \propto \frac{\exp[\delta \ln(aD/D_m - D)]^2}{D^4(D_m - D)} \quad (11)$$

where ($a = 1.26$ and $\delta = 1.13$). Limiting values of $\bar{K}/\rho_{32} = 0.549 \rho_{32}^2$ for ρ_{32} much less than 1, and $\bar{K}/\rho_{32} = 2.00/\rho_{32}$ for ρ_{32} much greater than 1 were tabulated using Equation (11). \bar{K} for $0.5 < \rho_{32} < 100$ for various m are tabulated in Appendix II. They were interpolated from the tables listed in Reference [2] for the values of m used in this report.

References [2], [4], [9], [13], [14], and [15] provide exact tabulation of the mean scattering coefficient vs various phaseshift parameters for a large variety of relative refractive indexes.

Another theory applicable to particle size determination is that for light scattering which will be presented briefly for the purpose of comparison. The intensity ratio for particles of any size illuminated by a monochromatic light beam of unit intensity is

$$I_0 = \frac{\lambda^2}{8\pi^2 R^2} \left[i_1(\theta) + i_2(\theta) \right] \quad (12)$$

where

I_0 is the intensity of light scattered at any angle
 θ is the angle between direction of incident beam and reverse direction of observed scattered light
 R is the distance from particle to point of observation
 $i_1(\theta)$ & $i_2(\theta)$ are functions of the angular distribution of light intensity proportioned to intensities of plane-polarized components of scattered light.

These last two terms, comprising the real complexity of

this theory, represent slowly converging series that are difficult to compute. Also, the measurement of scattered light at various distances and angles is difficult to accomplish without introducing variations in light intensity from stray light sources.

Light transmission theory was chosen for this experiment because of the ease with which it can be applied to any situation. An interference filter and a shield that allow only light passing through the test cell to enter the photomultiplier tube are all that is required for accurate measurements of particle size and concentration. Any stray light passing through the test cell would be measured by the photomultiplier tube as if it were from the laser beam. When particles are added to the test cell, the stray light intensity would be reduced by the same factor as that of the laser beam. Since both the stray light and the laser beam are reduced by the same factor, the transmission (I/I_0) will remain constant no matter how much stray light is present.

III. LIGHT TRANSMISSION EXPERIMENTAL APPARATUS

A major portion of the time spent on this project was devoted to the procurement and assembly of the components of the system shown in Figures 3 and 4. A detailed description of the apparatus is presented as an aid to those who continue experiments with this apparatus and as a means to help the reader better understand the experiment.

Photometer

The photometer was of central importance to this system, as it ultimately provided the data necessary to determine the size and concentration of the particles in question. Initial experiments were carried out with an Eldorado Model 210 Laboratory Differential Photometer which was equipped to measure the current output of two photomultiplier tubes. This piece of equipment proved to be very convenient when experimenting with scattering methods, as it was not necessary to take long intervals of time to move the photomultiplier tubes from position to position. This would have produced invalid results if the particles in question were changing size and/or concentration.

Since the bulk of the experiment was concerned with light transmission measurements, a newly acquired Pacific Photometric Model 11 Laboratory Photometer was used. Rapid change of photomultiplier tubes was enhanced by a high voltage control on this photometer that allowed the voltage to be varied from -500V to -1500V. In the Eldorado photometer, a rewiring of the internal high voltage circuits was necessary to meet the

requirements for a given photomultiplier tube. In addition to a variable high voltage power supply, the Model 11 further incorporated a wide range measuring circuit (necessary for measuring the greatly varying light intensities which occurred as particle concentrations increased) and an output for an external recorder. The instrument was designed for use with all side-window photomultiplier tubes and most head-on tubes having diameters of up to 2 inches.

The nine-position measuring circuit covered full scale ranges from 10 micro amperes to 1 nano ampere in $10 - 3 - 1$ steps. Zero adjustment of the meter and dark current cancellation assured accurate measurements. The current meter had three scales. Two of these scales were linear with 100 and 30 divisions, respectively. The third scale was the log of the reciprocal of the first scale and was a scale of optical density. Range adjustment permitted measurements from 100 per cent to 0.001 per cent on the linear scales. The overall accuracy of the measuring circuit was 2 per cent. A recorder output of 100mv for full scale on all ranges was provided on the back of the instrument, and was connected to a strip recorder. The regulation of the high voltage was 0.05 per cent for line variations from 105 to 125 volts AC. A constant voltage regulator attached to the Model 11 Photometer provided for the stabilization of the high voltage output.

Photomultiplier Tubes

Equally important were the photomultiplier tubes which converted incident light to some measurable quantity of current.

The photomultiplier tubes were ideal for measuring the low intensity light transmitted through a strong concentration of particles in a solution. The theory of photomultiplier tube operation is as follows. Radiation incident on the photoactive surface within the envelope of the tube will release an electron from the surface. A voltage drop then accelerates the electron to the first of several plates. Several secondary electrons are released when the electron strikes the first plate. These secondary electrons, in turn, are accelerated to another plate where the process continues. This chain reaction may be amplified, eventually, by as much as 10^6 times before the electrons are led from the photomultiplier tube to the photometer in the form of current. This current is the direct measure of light intensity sought for transmission measurements. Since the photomultiplier tubes are too sensitive for direct exposure to the laser light, with low particle concentrations, neutral density filters were used to reduce the laser intensity to one that could be measured. Reference [1], pages 791-793, lists seven special comments concerning the use of photomultiplier tubes that are especially useful to anyone not familiar with their operation or construction. While several photomultiplier tubes were used in preliminary measurements, an RCA 1P28 photomultiplier tube with good spectral responses for both wavelengths of lasers used in this experiment was used for the actual measurements. Figure 5 shows the spectral response of the 1P28 for various wavelengths of light. Light of wavelengths other than those of the lasers was prevented from affecting intensity measurements

by placing interference filters in front of the photomultiplier tubes. This permitted the use of normal working lights in the area of the experiment without affecting the normal intensity levels of the lasers.

In addition to the interference filters, a mirror and a beam splitter were used when both lasers were needed so that only one photomultiplier tube was required. (See Figure 4.) This arrangement enabled the two parallel laser beams to be superimposed on the same path after passing through the test cell. Thus, a single photomultiplier tube could accurately measure the intensity of either laser by alternately chopping off the light from the other laser.

Lasers

Two lasers were used for this experiment. A Spectra Physics Model 124 CW helium-neon gas laser of wavelength $\lambda = 6328 \text{ \AA}$ (See Figure 13) was used for particle size measurements when the concentration of the solution was known. This model consisted of two units. The laser unit contained the plasma tube, reflectors, supporting adjusting mechanisms, and the high voltage section of the excitation power supply, which supplied the energy necessary to create and sustain the glow discharge in the plasma tube. The separate exciter unit housed the low voltage exciter section located in the laser unit. To maintain the spectral purity of the red light that entered the photomultiplier tube, an interference filter of $\lambda = 6328 \pm 5 \text{ \AA}$ was used with this laser.

The second laser, used in conjunction with the first when neither particle size nor concentration was known, was a TRW Pulsed Argon Ion Laser Model 71A. (See Figure 14.) Although the laser's wavelength could be varied in six steps from 4579 Å to 5145 Å, 4880 Å was chosen because of the high peak power available at this wavelength. The TRW laser is a compact single unit that produces a repetitively pulsed argon ion laser light. The repetitive pulse rate can be varied from single pulse to 60 Hz (power line frequency). This variable repetitive pulse rate, properly timed with a light chopper on the continuous wave red laser, would permit alternate intensity measurements to be made for each laser without manually blocking off the light from each laser.

The advantage of using lasers for this experiment rather than the more commonly used white light is twofold. First, a laser produces a highly collimated beam of light of high intensity that enables the measurement of particle sizes even in very high concentrations of solutions. The limiting particle concentration is when multiple interference of the light occurs due to one particle shadowing another. This may be the reason for the deviation of the data from straight lines at high particle concentrations as seen in Figure 12. Second, the principal characteristic of the laser is that it produces a light of only one wavelength, thus eliminating the monochromator in the optical apparatus, shown in Figure 2. Since the laser beam is so highly collimated, a diverging lens and a collimating lens were used with

each laser to produce a broad parallel beam of light through the test cell. With a wider beam of light, inconsistencies in the concentration of the particles in solution tend to average out and give a more constant value for the transmitted light.

Instrumentation

In addition to the photomultiplier tube output meter in the photometer, a Leeds and Northrup Speedmax Strip Recorder provided a permanent record of the intensities measured by the photomultiplier tubes. (See Figure 6.) This record was especially helpful when the light from both lasers was alternately being measured. A variable DC power supply was also used to power the propeller in the test cell. The variable power output of the supply enabled the propeller speed to be adjusted as necessary to keep the particles in suspension. Figure 16 shows the arrangement of the instrumentation used for these experiments.

IV. LIGHT TRANSMISSION EXPERIMENTAL TECHNIQUE

Numerous tests were made to develop the transmission technique that was ultimately used. In each test, light transmitted through a variety of concentrations of particles was measured in order to insure that there existed an exponential variation of transmission with particle concentration. Initial measurements were made with one laser (the Spectra Physics Model 124) to establish procedures and techniques. Measurements using two lasers present little additional difficulty, once the technique is established with one laser.

In determining particle sizes of known concentrations, the following procedure was followed. Graphite particles were separated into groups of 0.281gm (equivalent of 0.125cc). The transmission apparatus was then warmed up for a minimum of 15 minutes. Twelve hundred milliliters (1200cc's) of water was poured into the test cell, and its temperature taken in order to establish its exact index of refraction. The red ($\lambda = 6328 \text{ \AA}$) laser—used in the tests on graphite particles— was aligned with the lenses and the photomultiplier tube, as shown in Figure 3. When the strip recorder was calibrated and the photometer was properly zeroed, the test was ready to begin. The light intensity of the laser, measured through the test cell with water, was recorded. Then samples of graphite particles were added one at a time, and the light intensity recorded after the addition of each sample. The test cell was then cleaned in preparation for the next test.

The graphite particles used were measured with a microscope. This was done at a comparison to the theorized particle size measurement. Stokes' Law provided another check on the actual particle size by measuring terminal velocity of particles as they sank in water. (See Appendix III.) Velocity measurement was provided by timing the fall of the particle over a fixed distance.

A settling process was used to provide a group of particles of similar size. Mixing a group of graphite particles in a test tube full of water and allowing them to settle gave a variation of particle sizes in the settled sediment, from small to large, i.e., from top to bottom. This scattering process is actually another application of Stokes' Law, implying that larger particles will fall faster than smaller ones. Then particle groups were siphoned off in three groups according to relative size.

To obtain experimental results for a group of particles of unknown size and concentration, an aerosol of ammonium-chloride particles was blown into the test cell. This test used vapors from flasks of ammonium hydroxide and of hydrochloric acid. These vapors were chosen because they remain a constant size long enough for the experiment to be completed (approximately 10 to 15 minutes) and because their size has been experimentally determined. [4]

The same experimental procedure was utilized as that for the case of unknown size, only with the following exception. Instead of adding graphite samples, more smoke was blown into the cell.

Intensity measurements were taken as soon as possible after the smoke was blown into the cell to insure that the particles had not begun to agglomerate. Once the variables necessary for the solution to the transmission equation were obtained, size determination for both types of particles was made from the tables of \bar{K}/ρ_{32} , shown in Appendix II. These tables were tabulated from computer solutions of Equations (4) and (9) for specific values of the relative indexes of refraction. Values of \bar{K} and \bar{K}/ρ_{32} for the values of m considered in this report were interpolated from the tables presented in Reference [2].

Since the ratio of light intensities passing through the test cell before and after the addition of particles is all that is necessary to determine the transmission, the units of intensity measurement are arbitrary. For these experiments the current produced by the light incident on the photomultiplier tube was used to determine the light transmission. Figure 6 is a record of the light transmissions recorded during experiment 12 by the Leeds and Northrup recorder.

Once the transmission was determined, Equation (7) was used to determine the mean particle diameter, D_{32} . The optical path length was a constant equal to the width of the test cell (4 cm).

For the first experiment, the volume concentration and the transmission were measured during the experiment. Figures 7 - show the graphical representation of the transmitted light for various amounts of graphite particles added to 1200cc of water.

The remaining unknown was the ratio of the mean scattering coefficient to the particle diameter (\bar{K}/D_{32}). Since $a \gg 1$ for these particles, the limiting value of \bar{K}/ρ_{32} , as given by Equation (10), was used to solve for the particle diameter.

For the second experiment, the concentration and the particle size of the aerosol of ammonium-chloride particles were unknown. However, by using two lasers of widely separated wavelengths and by measuring the transmission of each laser for the same concentrations, two equations with two unknowns could be written and solved for the mean particle size and concentration. Figure 17 shows this experiment in operation.

Table I is a sample of how the particle size was determined for experiment 12. Many different volumes of graphite particles were used to insure that each gave the same mean particle diameter. Table II lists the particle sizes measured by experiments 12 to 16.

Both procedures worked very satisfactorily, as evidenced by the comparison of the theoretical sizes to the sizes observed under magnification, and are recommended for future use.

V. RESULTS AND DISCUSSION

A major portion of the time and effort spent on this project was involved with the design and procurement of the apparatus used for the experiment. Procedures were established for the operation of the apparatus and are listed in checklist form in Appendix I.

As a check on the apparatus, a series of five tests on graphite particles in water were made. The particle sizes determined were compared with the sizes measured with the microscope. Another check of particle size was provided by Stokes' Law. (See Appendix III.) These five tests gave a mean particle diameter of 23.8μ with a maximum deviation of 0.5μ or 0.2 per cent. (Runs numbered 12 and 14 were not considered for this measurement since they represented extreme values of particle sizes. They were used to demonstrate the applicability of using a settling process to separate particle groups by size.) Microscopic measurements of particles from the same five groups showed diameters varying from 8.0μ for the smaller particles to 60.0μ for the largest particles. The variety of sizes shown under the microscope illustrated the value of light transmission measurements. Rather than try to precisely measure each particle in a large group of particles, light transmission experiments can be conducted to measure some mean size that describes the particles. Measurements using Stokes' Law gave a mean particle diameter of 50.0μ . A possible cause of the difference, here, is attributed to the difficulty of visually following the path of small particles over the measured drop in order to time their passage.

It can be seen from the above measurements that the theorized

mean diameter D_{32} is actually some diameter within the extreme boundaries of maximum and minimum particle sizes. Hence, it can be used accurately to give some measure of a middle diameter.

To test the light transmission theory for an unknown suspension, an aerosol of ammonium-chloride particles was pumped into the test cell. (See Figure 18.) Only two experiments were performed with this aerosol due to the late arrival of the TRW Pulsed Argon Ion Laser. However, results agreed quite closely with the measurements performed by Durbin [4].

Table III shows the data obtained from the first of two measurements on the ammonium-chloride aerosol. This experiment gave a mean particle diameter of 0.38μ as compared with Durbin's measurement (4) of 0.41μ .

Since the rocket exhaust to be tested by this transmission process was not developed prior to the presentation of this paper, no tests were performed. However Equation (7) shows that for the dynamic range (10^5) of the Model 11 Photometer, a minimum ratio of particle concentration to the phaseshift parameter, C_v/ρ_{32} , of 1.13×10^{-5} can be obtained.

It was noted from the light transmission measurements shown graphically in Figures 7 - 11 that the transmission values failed to follow a linear variation once a concentration of 0.00083cc graphite/cc water was reached. This nonlinearity was assumed to be due to a multiple scattering of light encountered when the particle concentration reached a certain value. Since the light transmission theory only holds for single scattering, particle

measurements for concentrations greater than 0.00083cc graphite/cc water are most likely invalid.

Figure 12 shows a plot of light transmission vs volume of graphite particles for three particle size measurements. Each of these tests was made for a different group of particles separated by the settling process described above. The variation of slopes shows that the particle sizes are different and that the bigger particles did sink faster. Thus, the settling process provided an excellent method for eliminating the extremes in particle sizes.

VI. CONCLUSIONS

The optical apparatus functioned satisfactorily, and procedures were established to make reliable operation of the equipment possible. The mean diameter of the graphite particles used was found to be 23.8μ , using the laser transmission technique. Microscope measurement of the same particles showed that this theorized mean diameter was within the extreme limits of particle size. The agreement between these measurements verified the light transmission theory used in this report.

Thus, the experiments performed have shown that the mean particle size theorized by Dobbins and Jimnagian [2] is closely related to the mean diameter of the size distribution function. Hence, the mean diameter measured by a light transmission experiment provided some indication of the particle sizes present in the suspension. For the special case of uniform particle sizes, the light transmission theory would provide an exact measurement of the size.

Finally, mean scattering coefficients were interpolated for the solutions used. These coefficients were tabulated for the relative indexes of refraction of graphite in water and of an aerosol of ammonium-chloride particles. (See Appendix II)

VII. SUGGESTIONS FOR FURTHER INVESTIGATION

The light transmission used for these experiments provides a very flexible test for determining particle sizes and concentrations in known suspensions. Current ideas for investigation include:

- a. Make measurements of particle sizes on the order of one milli-micron to determine what range of particle sizes can be accurately measured.
- b. Conduct practical experiments to determine the applicability of using the light transmission theory to measure particle sizes and concentrations in fogs, exhausts, and smogs.
- c. Incorporate a light chopper on the continuous wave laser, and regulate the pulsed laser to permit nearly simultaneous light intensity measurements in order to reduce any errors which may enter due to the growth of particles during experimentation.

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TABLE I
SAMPLE DATA SHEET

RUN # 12 $I_0 = 91.0$

VOLUME OF GRAPHITE cc	INTENSITY I	TRANSMISSION T	$-\ln T$	$C_v \times 4.008 \times 10^5$	\bar{K}/ρ_{32}
0.125	53.0	0.582	0.54	41.75	.0130
0.250	31.0	0.340	1.07	83.42	.0142
0.375	19.0	0.208	1.56	125.25	.0142
0.500	11.0	0.120	2.11	166.99	.0145
0.625	6.8	0.074	2.59	208.74	.0144
0.750	4.4	0.048	3.22	250.50	.0142
0.875	2.8	0.030	3.48	292.25	.0142
1.000	1.7	0.018	3.98	333.99	.0137
1.125	1.0	0.019	4.52	375.75	.0133
1.250	0.67	0.007	4.92	417.50	.0130
avg.					.01425

For $\bar{K}/\rho_{32} = 0.01425$

$\rho_{32} = 140.20$

$D_{32} = 2.1 \times 10^{-3} \text{ cm}$

$= 0.00082 \text{ in}$

RUN # 12

Microscopic measurement shows:

$0.002 \text{ in} < D_{32} < 0.0002 \text{ in}$

TABLE II
GRAPHITE PARTICLE SIZE MEASUREMENTS

RUN NO.	MEASURED SIZE - cm
12	0.00210
13	0.00239
14	0.00285
15	0.00242
16	0.00233

Mean Particle Size

0.00242 cm

Maximum Deviation

0.00043 cm or 17.7 % *

* Note: Runs 12, 13, & 14 were made on particles sorted as to size by a settling process in water, with the particles then grouped as small, medium, and large, respectively. Hence, the maximum deviation calculated has little value since it is calculated for extremes rather than for a "normal" distribution of particle sizes.

TABLE III
DATA SHEET FOR THE FIRST AMMONIUM-CHLORIDE AEROSOL EXPERIMENT

T_r	$-\ln T_r$	T_b	$-\ln T_b$	$C_v(\bar{K}/\rho_{32})_r \times 10^5$	$C_v(\bar{K}/\rho_{32})_b \times 10^5$	$C_v \times 10^6$
0.52	0.73	0.35	1.04	0.192	0.208	2.7
0.33	1.10	0.21	1.57	0.286	0.312	3.3
0.11	2.21	0.047	3.14	0.575	0.623	6.1
0.068	2.75	0.015	4.19	0.716	0.831	8.2
0.0083	4.79	0.0011	6.80	1.247	1.206	13.5

41

FROM APPENDIX II

for;

$$(\bar{K}/\rho_{32})_r = 0.763 \quad \rho_{32} = 2.42$$

$$(\bar{K}/\rho_{32})_b = 1.039 \quad \rho_{32} = 3.14$$

$$\text{from } \rho_{32} = 2.42 \quad D_{32} = \frac{6328 \times 10^{-8} (2.42)}{2 (1.642 - 1)} = 0.379 \mu$$

$$\rho_{32} = 3.14 \quad D_{32} = \frac{4880 \times 10^{-8} (3.14)}{2 (1.642 - 1)} = 0.380 \mu$$

Note: Subscripts (r and b) indicate use of red ($\lambda = 6328 \text{ \AA}$) or blue-green ($\lambda = 4880 \text{ \AA}$) laser.

- A. High-voltage photocell power supply
- B. RCA IP21 multiplier phototube
- C. Ballantine model 304 VTVM
- D. Chamber for generating ammonium-chloride fog
- E. Glass windows
- F. Wratten light-filter monochromat 77A
- G. Focusing lens
- H. Water infrared filter
- I. Adjustable slit
- J. Air-cooled, high pressure mercury-vapor lamp (GE B-H6)
- K. Lamp power supply

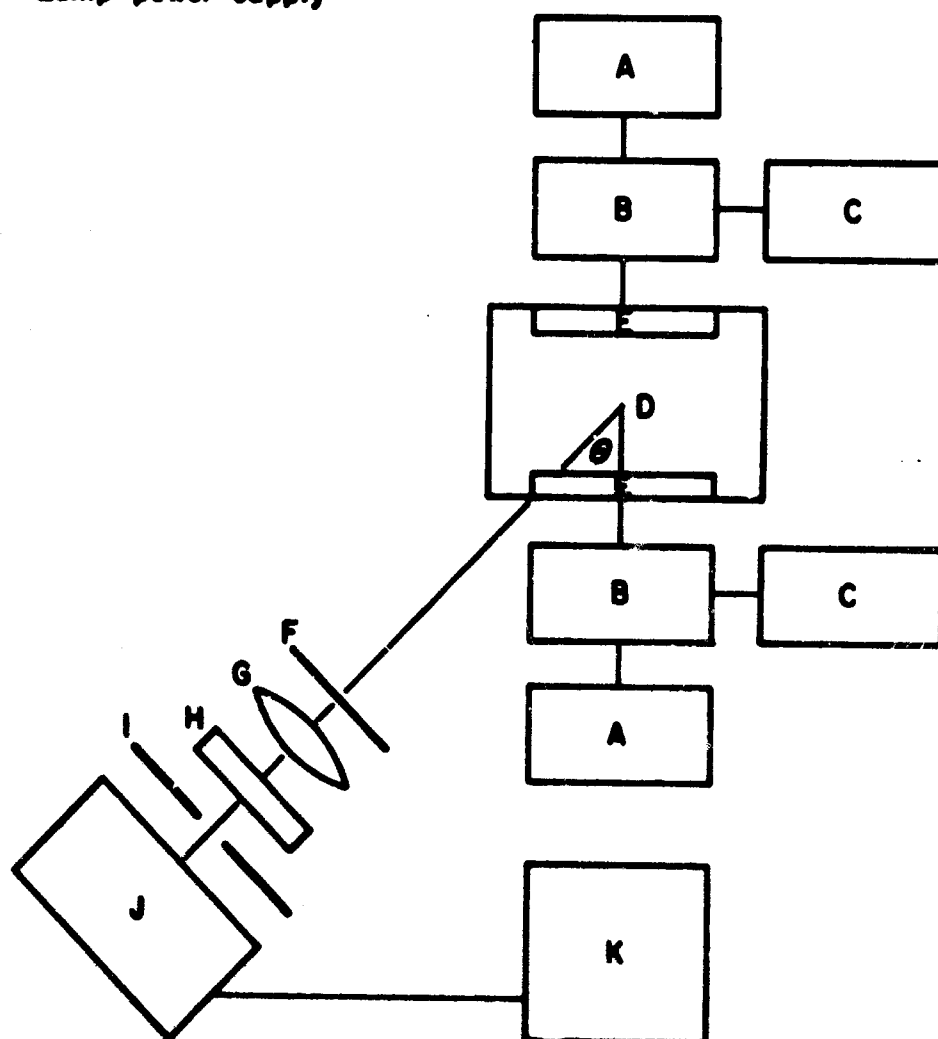


FIG. 1 - EXPERIMENTAL ARRANGEMENT USED BY DURBIN TO MEASURE PARTICLE SIZES IN AMMONIUM-CHLORIDE FOG BY LIGHT-SCATTERING METHODS [4]

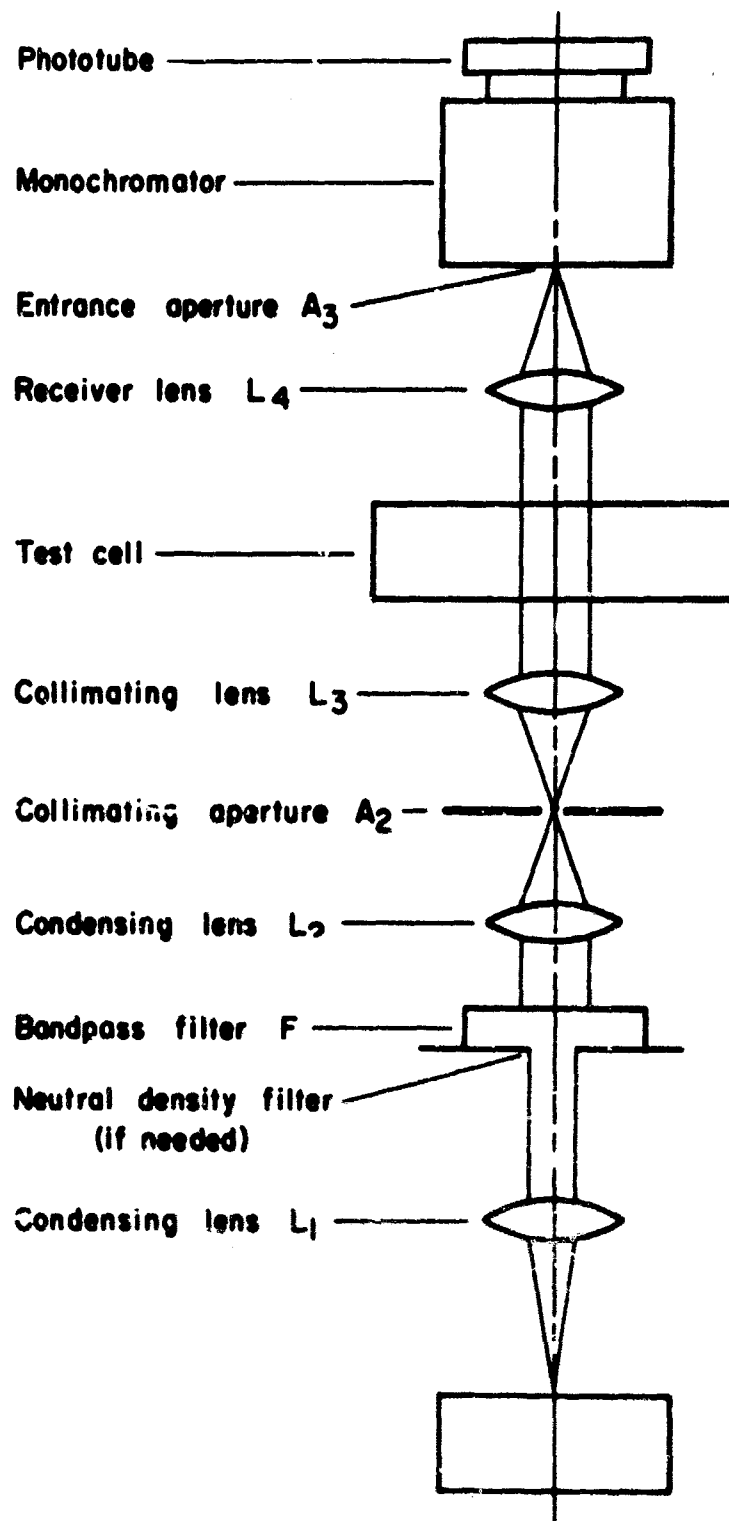


FIG. 2 - SCHEMATIC DRAWING OF OPTICAL APPARATUS USED BY DOBBINS AND JIZMAGIAN FOR LIGHT TRANSMISSION MEASUREMENTS [2]

- A. Spectra-Physics model 124
stabilite gas laser $\lambda = 6328 \text{ \AA}$
- L₁ Condensing lens
- L₂ Collimating lens
- B. Test cell
- C. Interference filter
- D. RCA IP28 photomultiplier tube
- E. PPI laboratory photometer
- F. Leeds & Northrup speedomax
strip recorder
- G. D.C. power supply

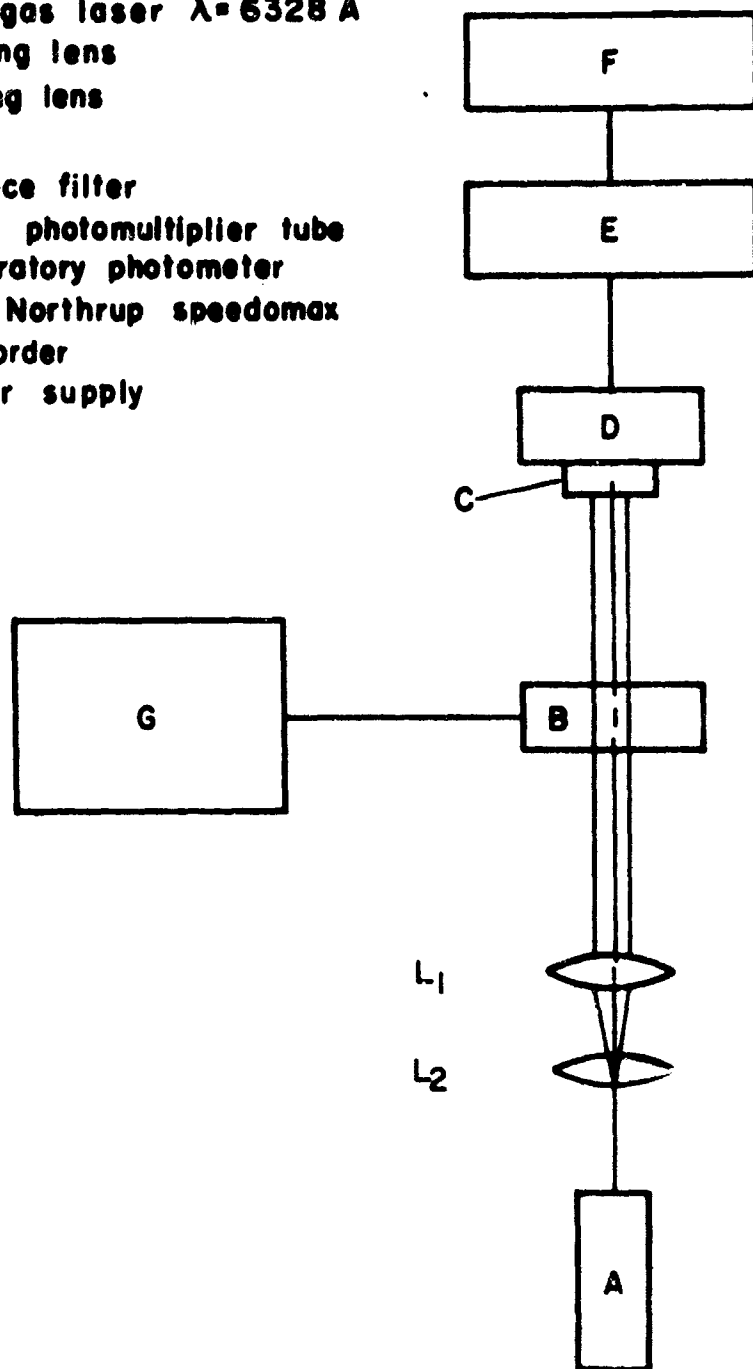


FIG. 3 - OPTICAL APPARATUS USED FOR LIGHT TRANSMISSION MEASUREMENTS ON SOLUTIONS OF KNOWN CONCENTRATION BUT UNKNOWN SIZE

- A. Spectra-Physics model 124
stabilite gas laser $\lambda = 6328 \text{ \AA}$
- B. TRW pulse argon ion laser
 $\lambda = 4880 \text{ \AA}$
- L₁ Condensing lenses
- L₂ Collimating lenses
- C. Test cell
- D. Interference filters
- M. Mirror
- E. Beam splitter
- F. RCA 1P28 photomultiplier tube
- G. PPI laboratory photometer
- H. Leeds & Northrup speedomax
strip recorder

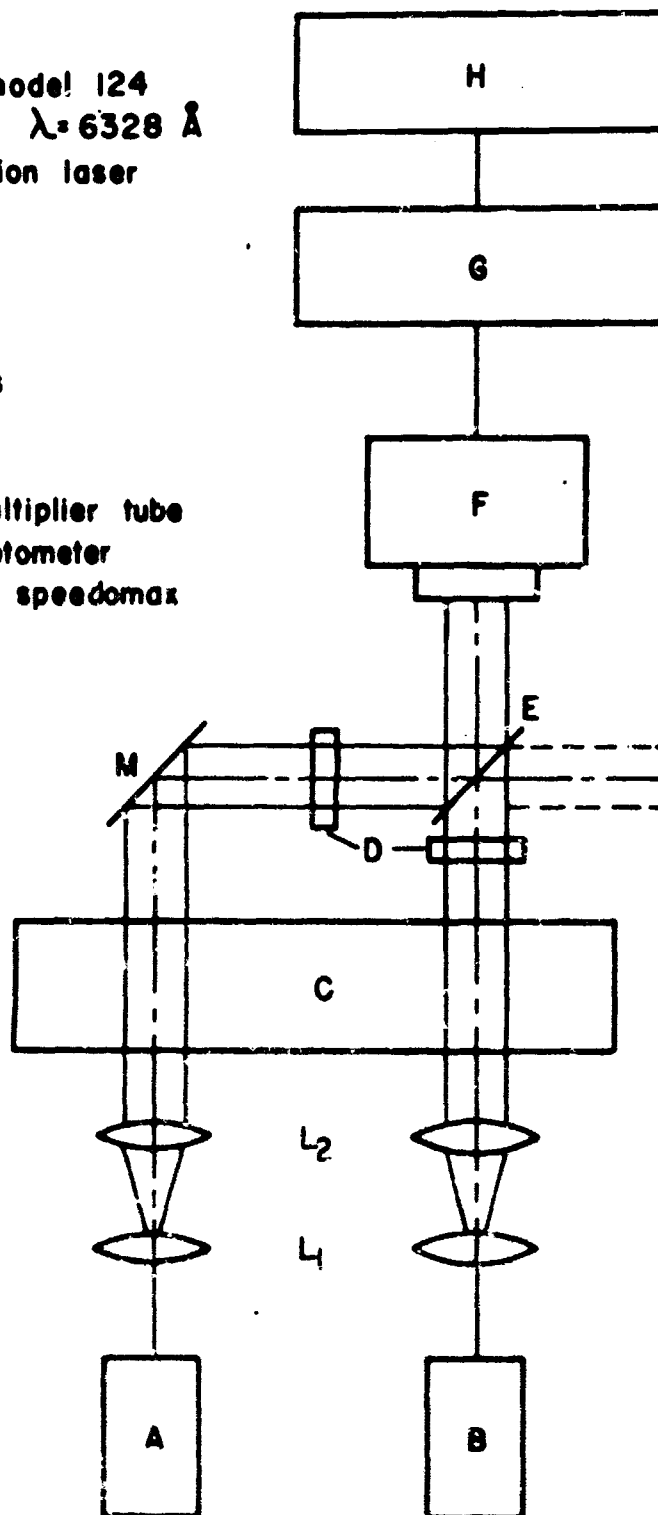


FIG. 4 OPTICAL APPARATUS USED FOR LIGHT TRANSMISSION MEASUREMENTS ON SOLUTIONS OF UNKNOWN SIZE AND CONCENTRATION

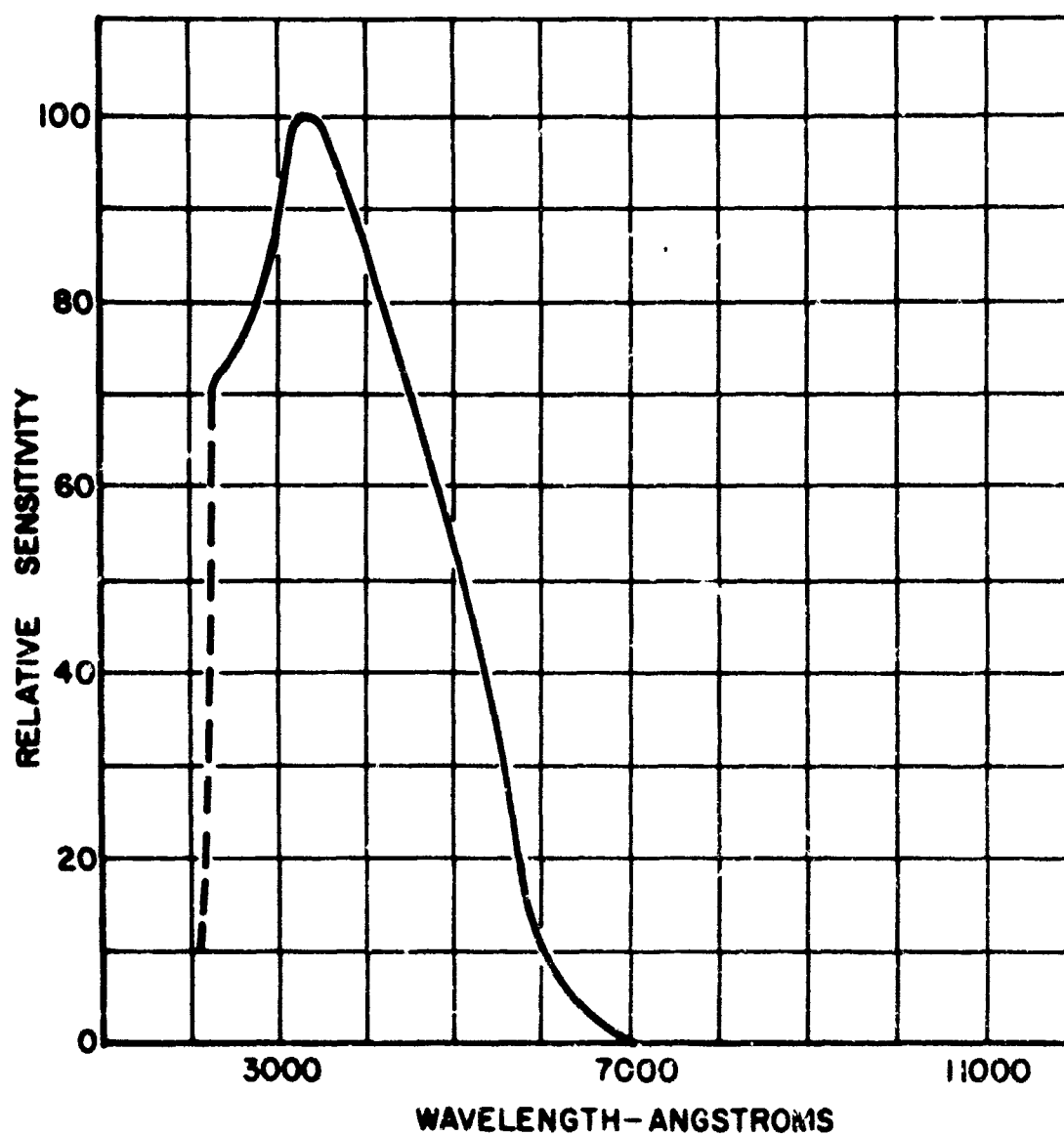
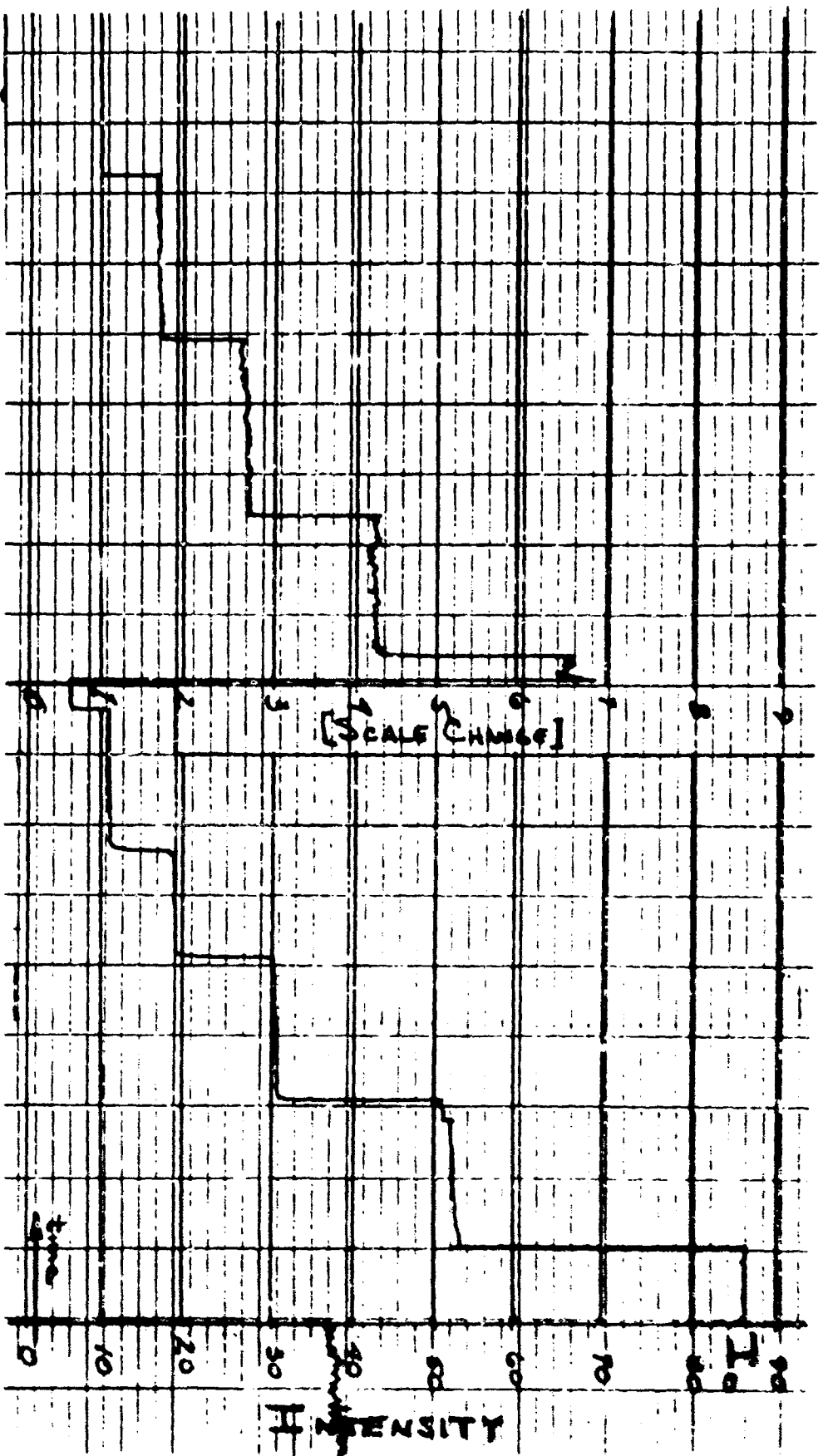


FIG. 5 SPECTRAL RESPONSE OF RCA IP29
PHOTOMULTIPLIER TUBE

FIG. 6 - DEPENDENCE OF INTENSITY ON CONCENTRATION OF PARTICLES
 (Steps indicate the addition of 0.125 cc of graphite particles to water)



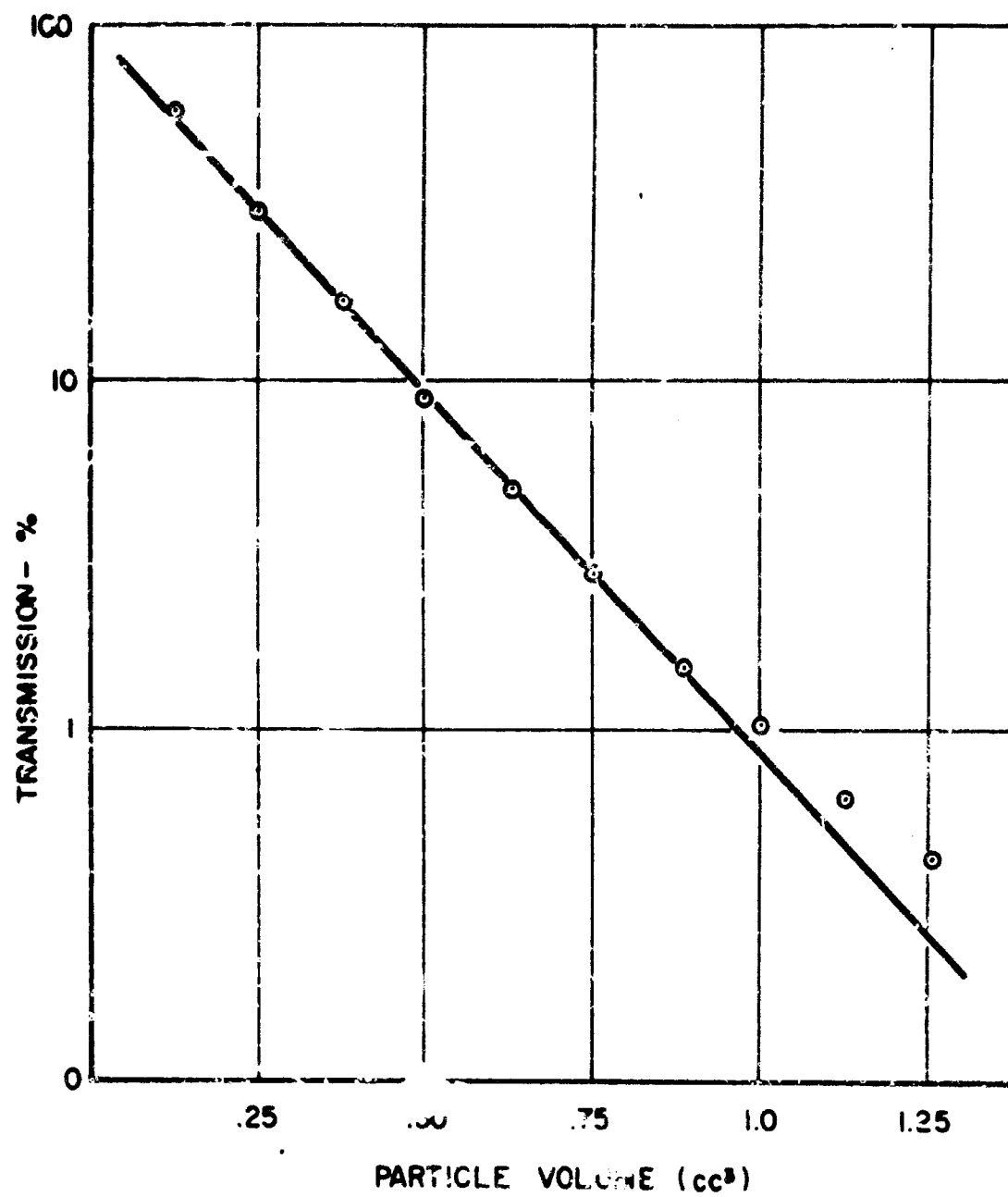


FIG 7 GRAPHITE PARTICLE MEASUREMENT - RUN 12

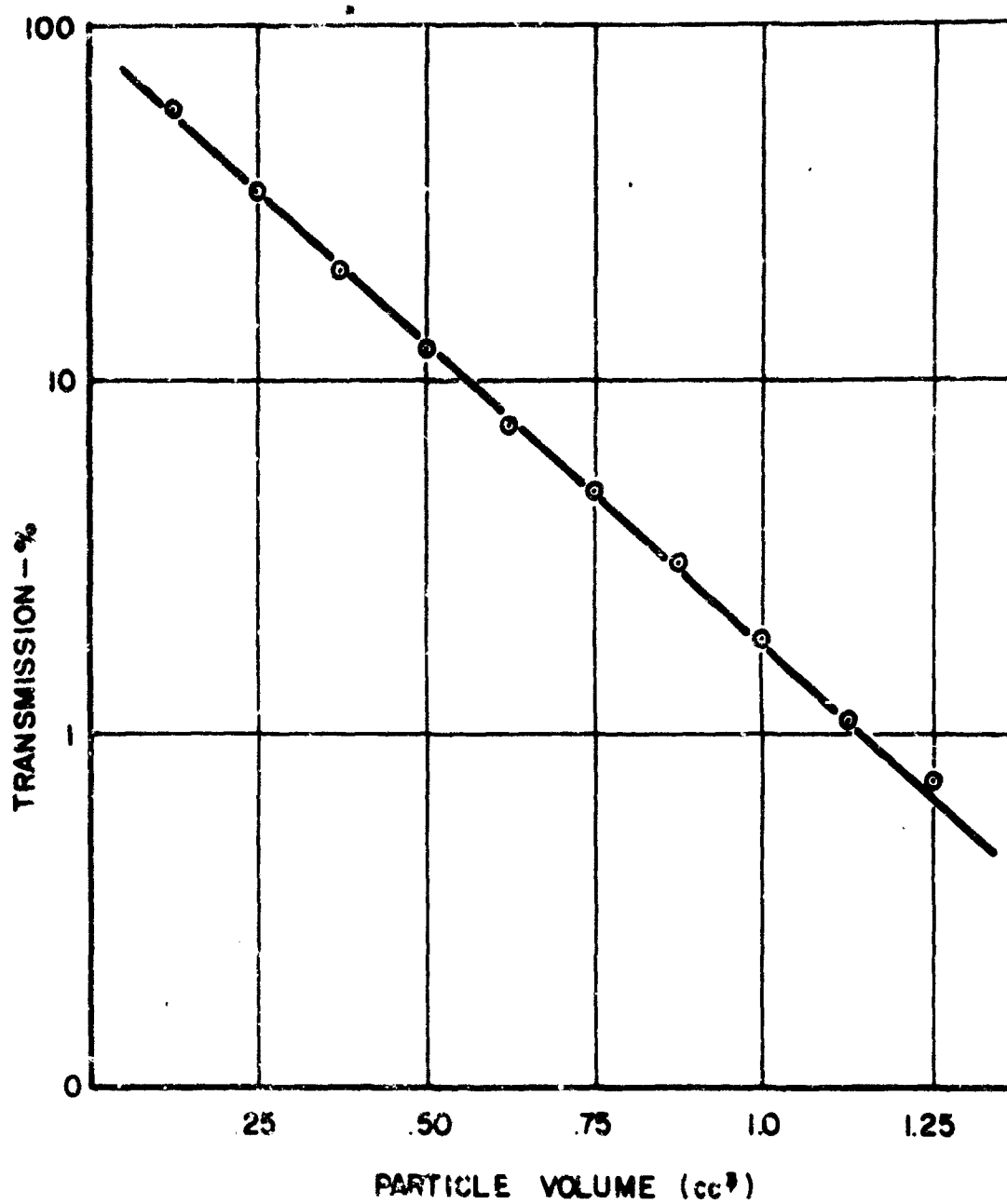


FIG. 8 GRAPHITE PARTICLE MEASUREMENT— RUN 13

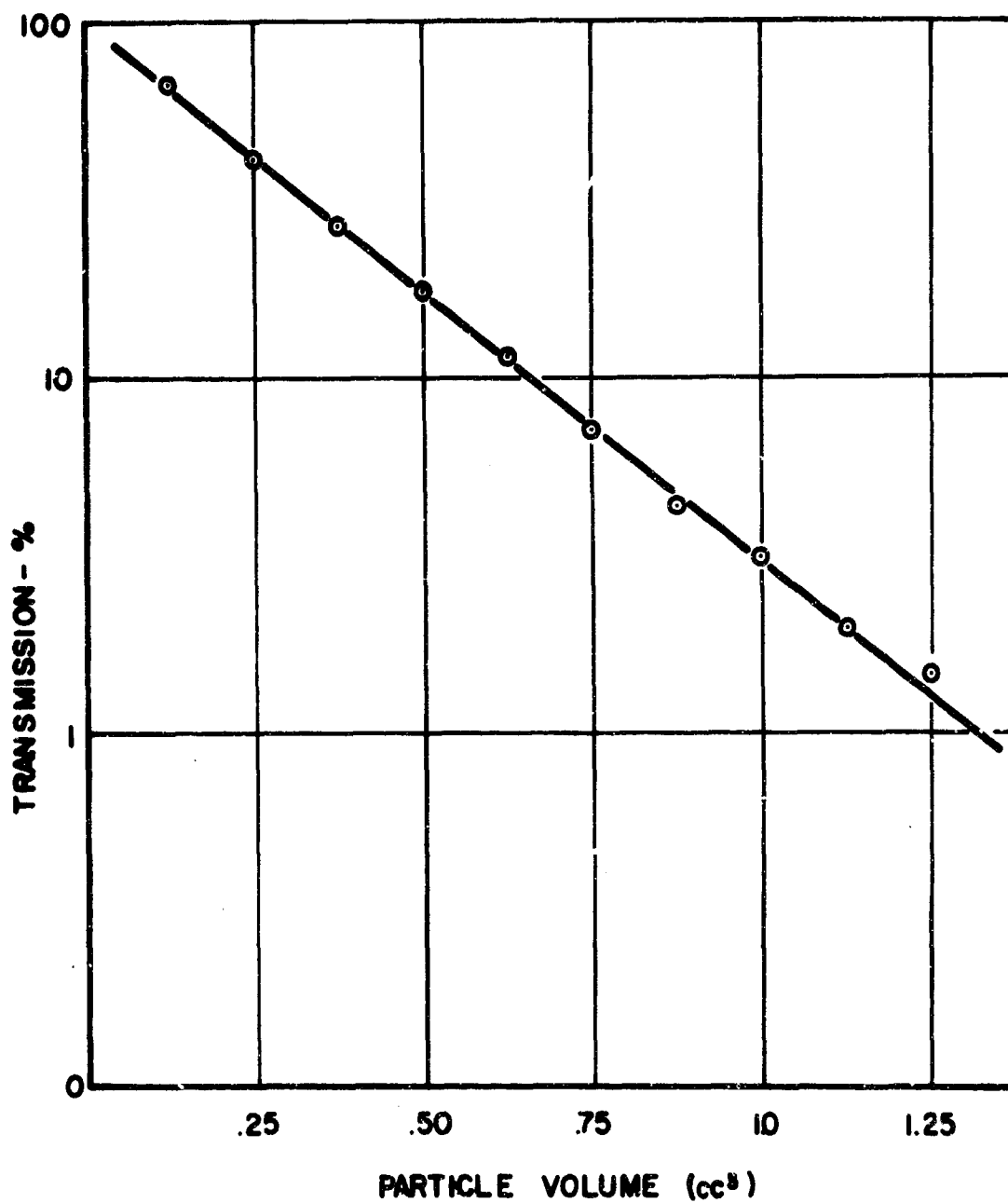


FIG. 9 GRAPHITE PARTICLE MEASUREMENT — RUN 14

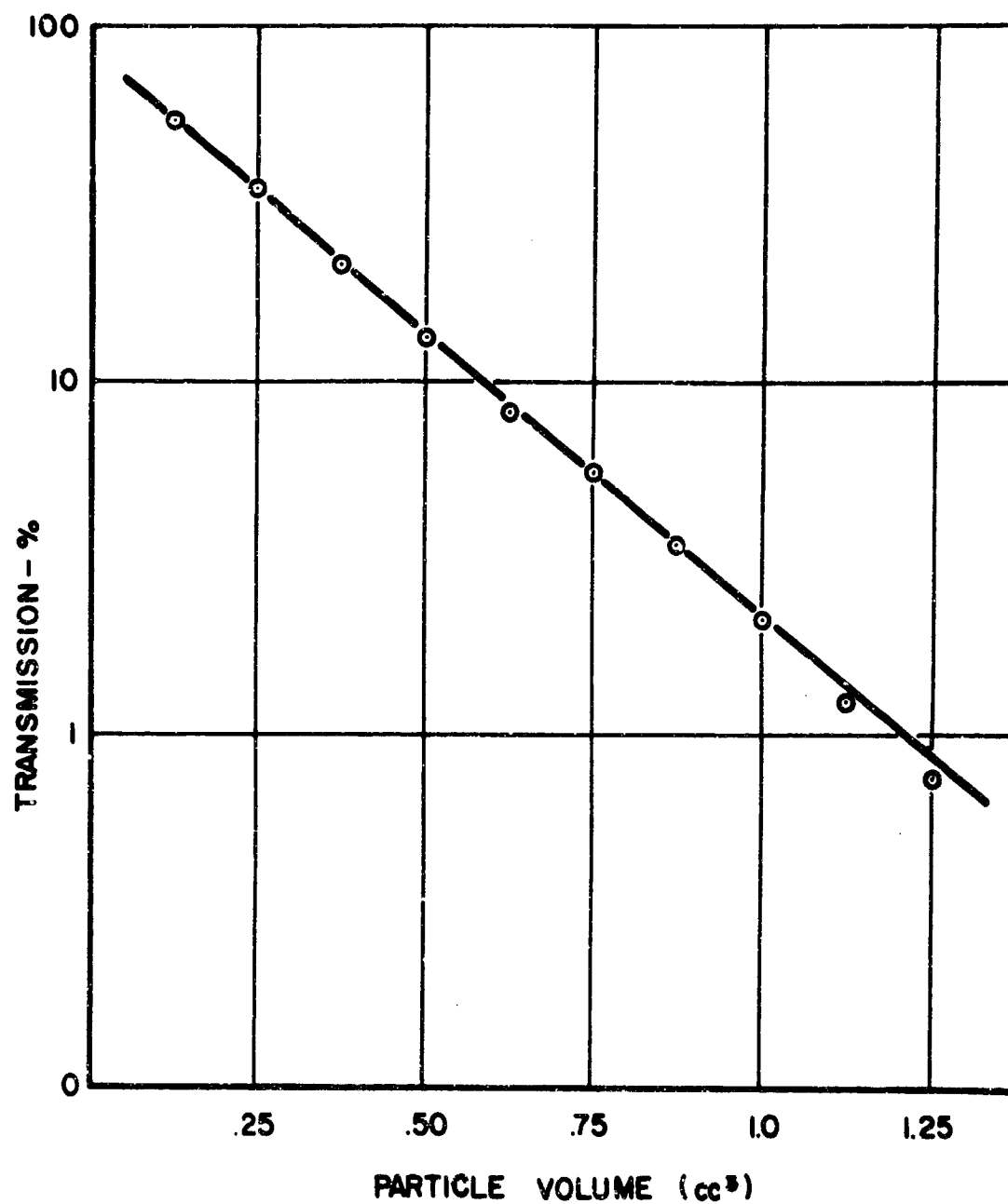


FIG.10 GRAPHITE PARTICLE MEASUREMENT — RUN 15

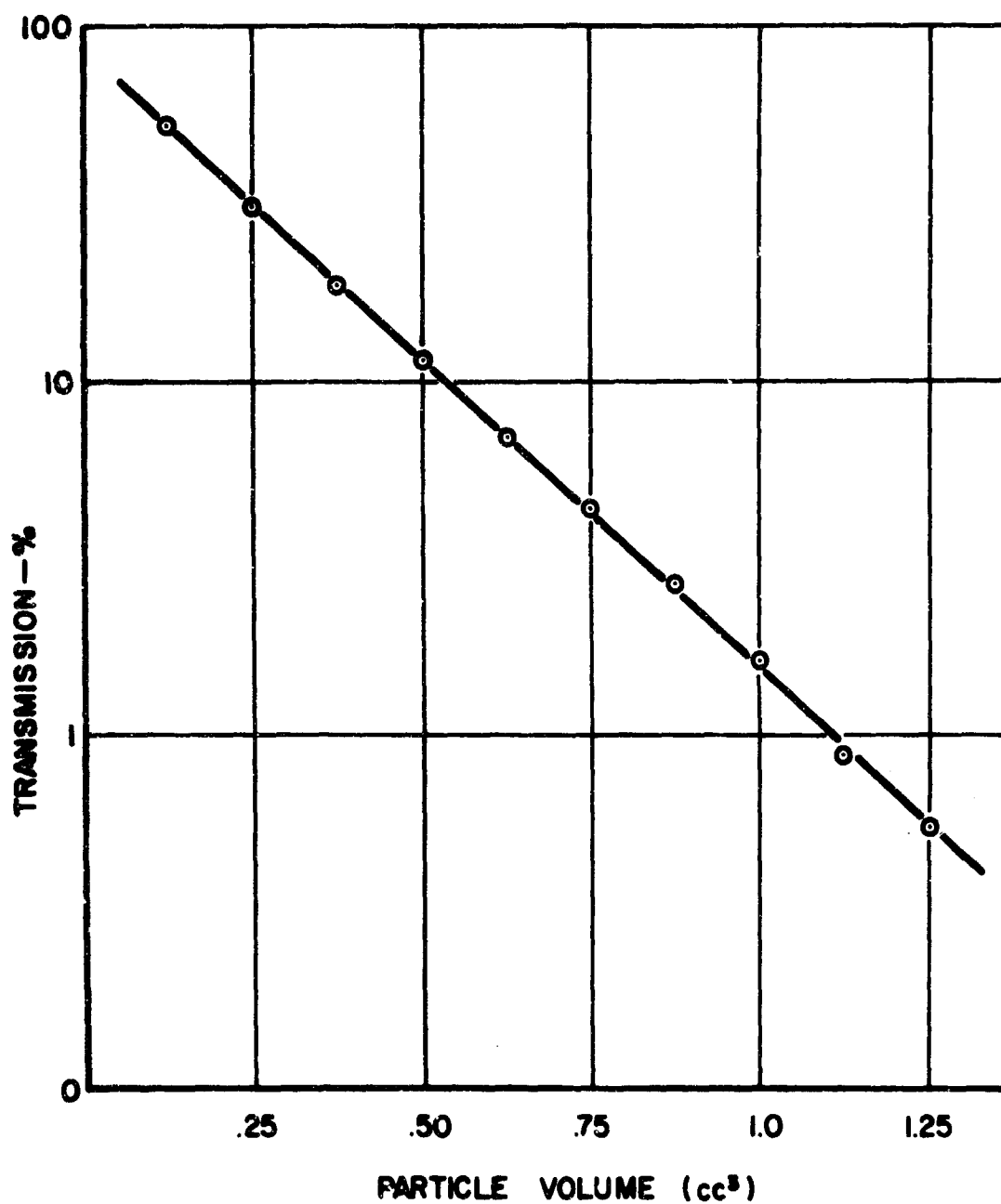


FIG. 11 GRAPHITE PARTICLE MEASUREMENT — RUN 16

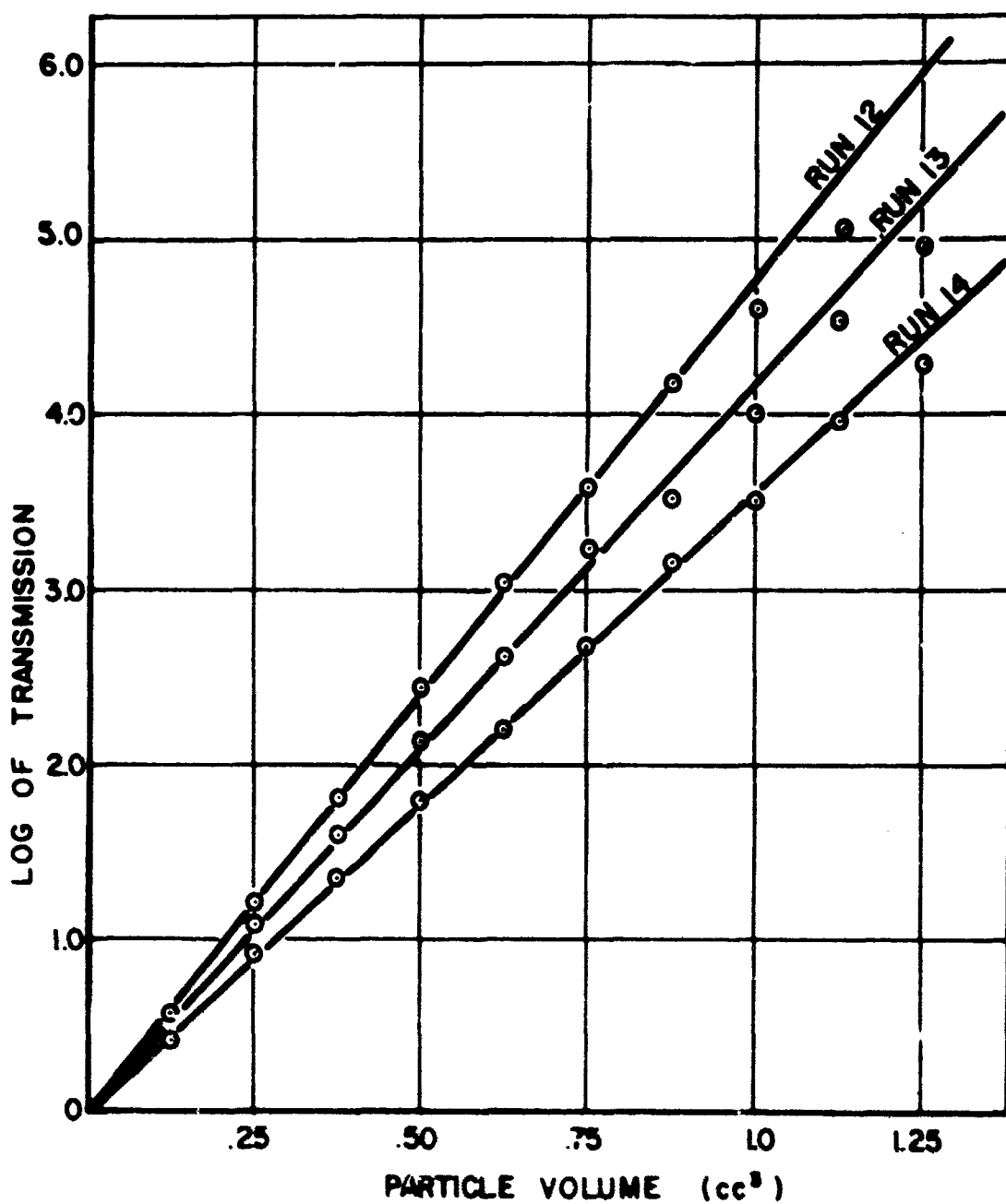


FIG.12 - TRANSMISSION MEASUREMENT OF 3 GROUPS OF CARBON PARTICLES THAT HAVE BEEN SORTED BY SIZE IN A SETTLING PROCESS

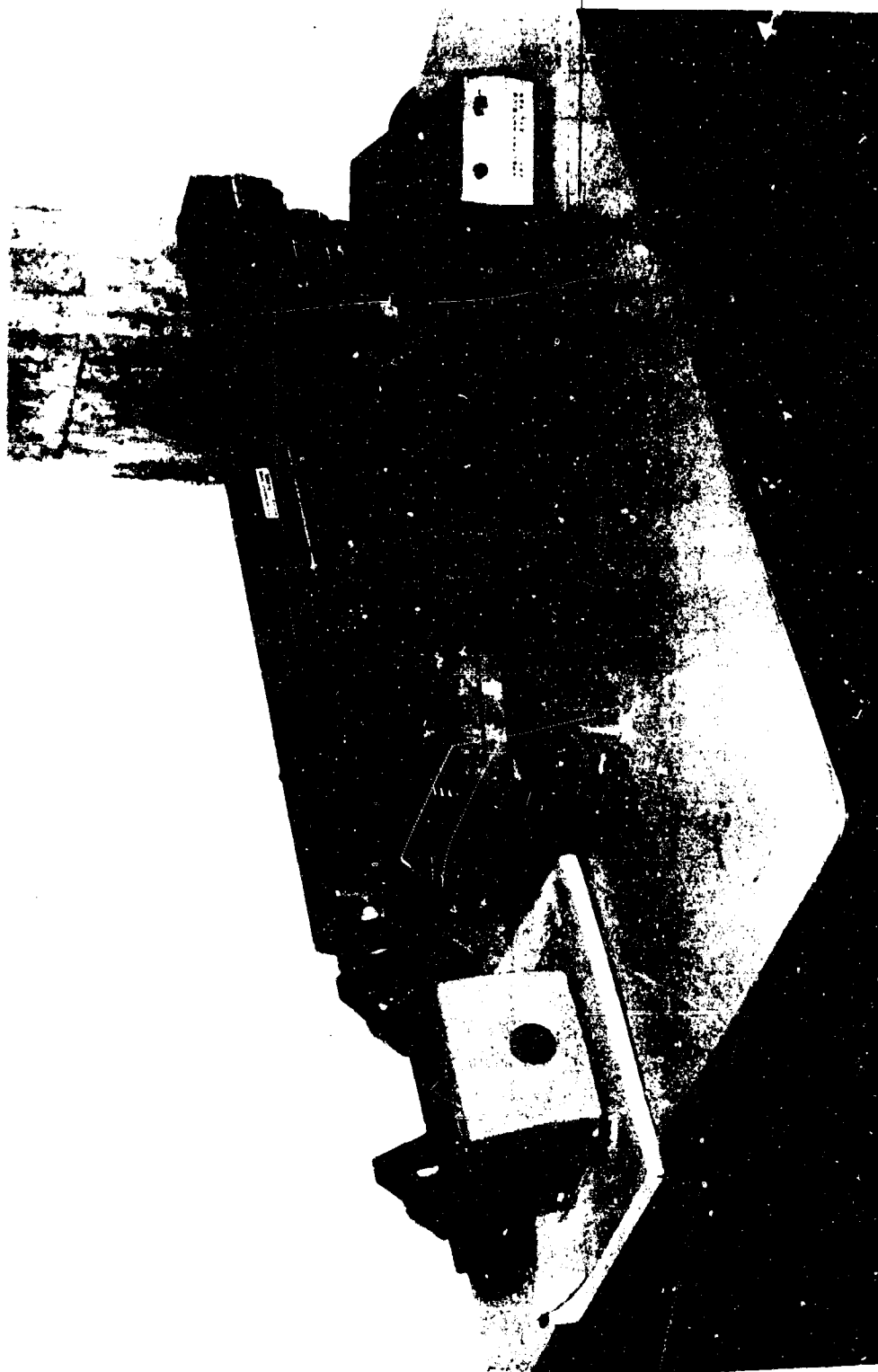


FIG.13 -SPECTRA PHYSICS MODEL-124 CONTINUOUS WAVE LASER WITH
DIVERGING AND COLLIMATING LENSES

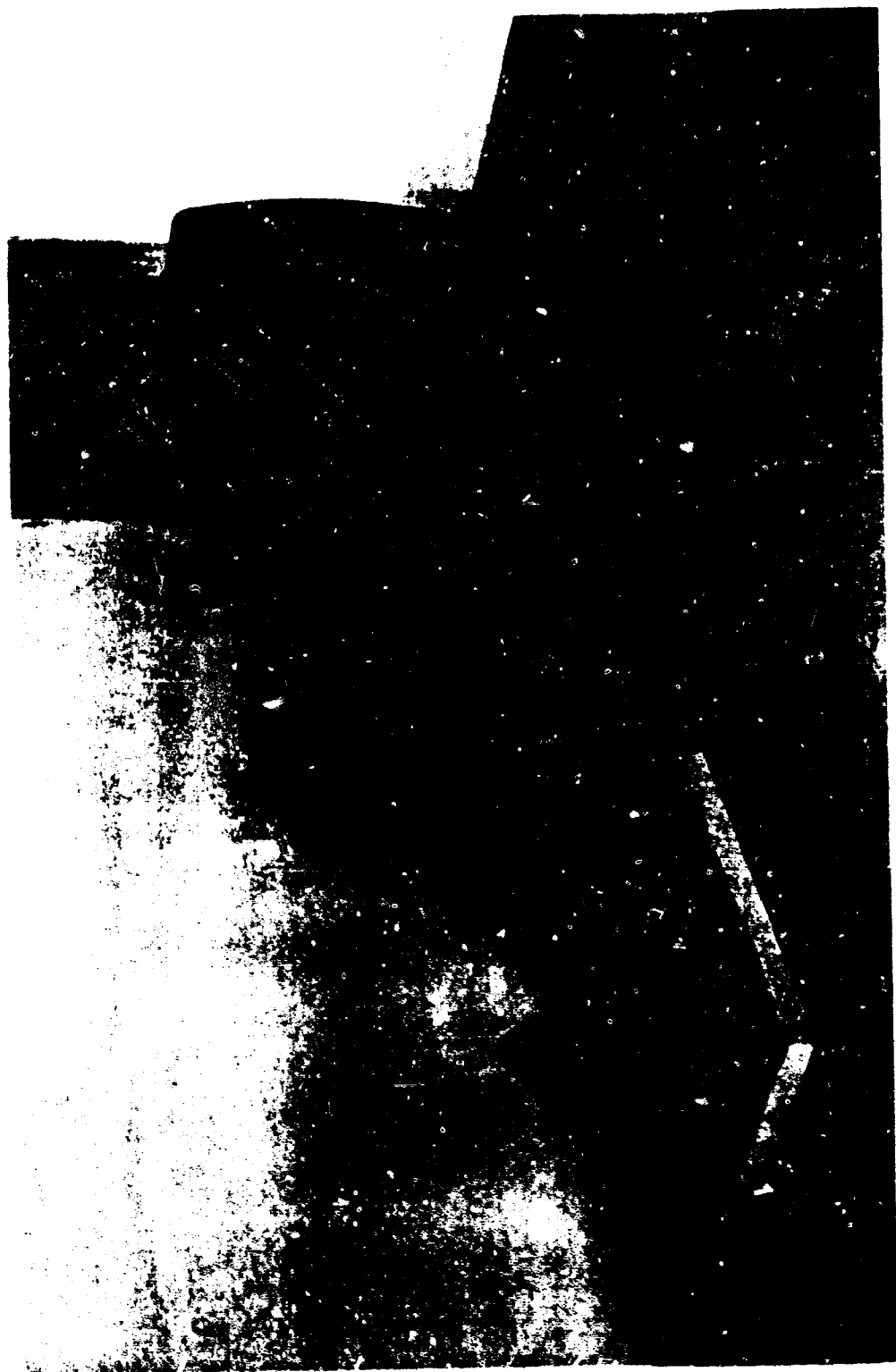


FIG. 4 -TRW PULSE ARGON-ION LASER WITH DIVERGING AND COLLI-
MATING LENSES

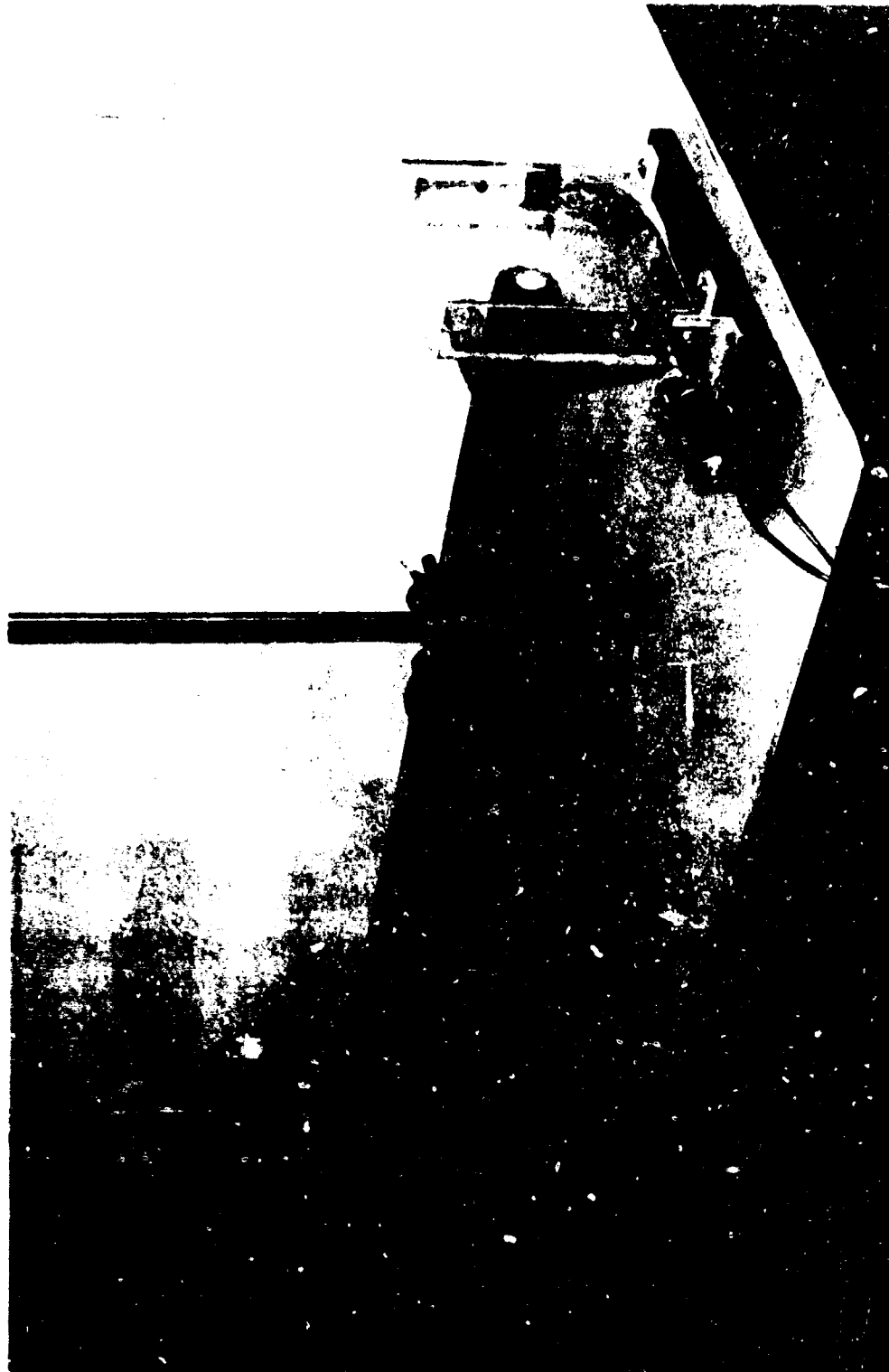


FIG.15 -PHOTOMULTIPLIER AND TEST CELL



FIG. 16 - INSTRUMENTATION



FIG.17 -EQUIPMENT IN OPERATION



FIG. 8 -TEST CELL EQUIPPED TO GENERATE GASEOUS SUSPENSIONS

APPENDIX I

PROCEDURES CHECKLIST

Preparation for Experiment

1. Measure 0.281 gm (0.125 cc) samples of Graphite.
2. Measure 1200 ml (1200 cc) of water in a beaker.
3. Clean test cell.
4. Clean lenses, filters, and mirror of optical apparatus.
5. Align laser beam, lenses, test cell, and photomultiplier.
6. Start laser.
7. Turn strip recorder ON.
8. Turn photometer power toggle switch ON.
9. Turn high voltage toggle switch ON.
10. Turn meter display switch to H. V., and adjust high voltage to some value less than the maximum rating for the photomultiplier in use minus 55 V.

Test Procedure

1. Turn meter display switch to INPUT.
2. Turn sensitivity switch OFF.
3. Cover photomultiplier tube.
4. Turn zero adjust control until meter reading is 0.
5. Uncover photomultiplier tube and expose it to laser beam passing through test cell.
6. Turn input current switch to scale that gives a current reading greater than 10 per cent of full scale.
7. Turn on strip recorder.
8. Start Prop motor.
9. Record meter reading for laser passing through water only.
10. Add Graphite sample.
11. Record meter reading.
12. Turn OFF prop motor.
13. Turn recorder OFF.
14. Turn H. V. and power switches OFF.
15. Turn laser off.

Note 1: Steps 10 and 11 are repeated as many times as measurements of particle size are made for different particle concentrations.

Note 2: When two lasers are in use, measurements and recordings should be made by alternately blocking off the light from one laser to the photomultiplier and then from the other laser.

APPENDIX II

MEAN SCATTERING COEFFICIENT vs PHASE SHIFT PARAMETER

\bar{K} vs ρ_{32} for upper limit function ($a = 1.130$, $\delta = 1.260$)

for selected values of m interpolated from Reference [2]

	$M = 1.65$	$M = 1.673$	$M = 1.75$
ρ_{32}	\bar{K}	\bar{K}	\bar{K}
0.500000	0.01297	0.01208	0.00910
1.000000	0.19570	0.18528	0.15040
1.500000	0.76880	0.74341	0.65840
2.000000	1.62100	1.59846	1.52300
2.500000	2.41500	2.46074	2.41300
3.000000	3.15000	3.14655	3.13500
3.500000	3.77500	3.74637	3.73500
4.000000	4.30000	4.27203	4.27500
4.500000	4.72000	4.74032	4.70500
5.000000	5.05000	5.17063	5.23300
5.500000	5.28000	5.52102	5.57800
6.000000	5.42000	5.78187	5.83500
6.500000	5.47000	5.98972	5.93900
7.000000	5.43000	6.12380	6.07000
7.500000	5.30000	6.19465	6.17700
8.000000	5.08000	6.21150	6.25100
8.500000	4.78000	6.17235	6.29700
9.000000	4.42000	6.08495	6.24100
9.500000	4.00000	5.95444	6.17800
10.000000	3.53000	5.7838	6.06200
10.500000	3.00000	5.57730	5.91790
11.000000	2.42000		5.75000

	$M = 1.65$	$M = 1.673$	$M = 1.75$
ρ_{32}	R/ρ_{32}	R/ρ_{32}	R/ρ_{32}
0.500000	0.02594	0.02416	0.01820
1.000000	0.19570	0.18528	0.15040
1.500000	0.76880	0.74341	0.65840
2.000000	1.62100	1.59846	1.52300
2.500000	2.41500	2.46074	2.41300
3.000000	3.15000	3.14655	3.13500
3.500000	3.77500	3.74637	3.73500
4.000000	4.30000	4.27203	4.27500
4.500000	4.72000	4.74032	4.70500
5.000000	5.05000	5.17063	5.23300
5.500000	5.28000	5.52102	5.57800
6.000000	5.42000	5.78187	5.83500
6.500000	5.47000	5.98972	5.93900
7.000000	5.43000	6.12380	6.07000
7.500000	5.30000	6.19465	6.17700
8.000000	5.08000	6.21150	6.25100
8.500000	4.78000	6.17235	6.29700
9.000000	4.42000	6.08495	6.24100
9.500000	4.00000	5.95444	6.17800
10.000000	3.53000	5.7838	6.06200
10.500000	3.00000	5.57730	5.91790
11.000000	2.42000		5.75000

Note: Columns 2 and 4 are exact tabulations of the mean scattering coefficient. Column 3 is the interpolated value applicable to a solution of Carbon in water.

\bar{K} vs p_{02} for upper limit function ($a = 1.130, b = 1.260$) for selected values of M interpolated from Reference [2]

	M=1.50	M=1.642	M=1.65
RFO 32	\bar{K}	\bar{K}	\bar{K}
0.50000	0.02336	0.01352	0.01297
1.00000	0.28210	0.28210	0.28210
1.50000	0.91960	0.77684	0.76880
2.00000	1.72600	1.62660	1.62100
2.50000	2.47900	2.47521	2.47500
3.00000	3.04100	3.14419	3.15000
3.50000	3.36000	3.55123	3.56200
4.00000	3.45800	3.70319	3.71700
5.00000	3.24900	3.50649	3.52100
6.00000	2.93000	3.14016	3.15200
7.00000	2.72500	2.89445	2.90400
8.00000	2.61200	2.75779	2.76600
9.00000	2.53800	2.66769	2.67500
10.00000	2.48600	2.60339	2.61000
12.00000	2.42000	2.51656	2.52200
14.00000	2.36300	2.45483	2.46000
16.00000	2.33000	2.40763	2.41200
18.00000	2.30200	2.36921	2.37300
20.00000	2.27900	2.33769	2.34100
30.00000	2.19500	2.23192	2.23400
50.00000	2.11700	2.17001	2.17300
100.00000	2.05900	2.07415	2.07500
	M=1.50	M=1.642	M=1.65
p_{02}	R/p_{02}	R/p_{02}	R/p_{02}
RFO 32	RATIO	RATIO	RATIO
0.50000	0.04672	0.02705	0.02594
1.00000	0.28210	0.28210	0.28210
1.50000	0.61307	0.51790	0.51253
2.00000	0.86300	0.81330	0.81050
2.50000	0.99160	0.99008	0.99000
3.00000	1.01367	1.04806	1.05000
3.50000	0.96000	1.01464	1.01771
4.00000	0.86450	0.92580	0.92925
5.00000	0.64980	0.70130	0.70420
6.00000	0.48833	0.52336	0.52533
7.00000	0.38929	0.41349	0.41486
8.00000	0.32650	0.34472	0.34575
9.00000	0.28200	0.29641	0.29722
10.00000	0.24860	0.26034	0.26100
12.00000	0.20167	0.20971	0.21017
14.00000	0.16879	0.17534	0.17571
16.00000	0.14562	0.15048	0.15075
18.00000	0.12789	0.13162	0.13183
20.00000	0.11395	0.11688	0.11705
30.00000	0.07317	0.07440	0.07447
50.00000	0.04234	0.04340	0.04346
100.00000	0.02059	0.02074	0.02075

m = 1.642

\bar{p}_{32}

\bar{k}

\bar{k}/\bar{p}_{32}

0.55000
0.60000
0.65000
0.70000
0.75000
0.80000
0.85000
0.90000
0.95000
1.00000
1.05000
1.10000
1.15000
1.20000
1.25000
1.30000
1.35000
1.40000
1.45000
1.50000
1.55000
1.60000
1.65000
1.70000
1.75000
1.80000
1.85000
1.90000
1.95000
2.00000
2.05000
2.10000
2.15000
2.20000
2.25000
2.30000
2.35000
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2.50000
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3.95000
4.00000
4.10000
4.20000
4.30000
4.40000
4.50000
4.60000
4.70000
4.80000
4.90000
5.00000

0.04038
0.06724
0.09410
0.12095
0.14781
0.17467
0.20153
0.22838
0.25524
0.28210
0.33157
0.38105
0.43052
0.48000
0.52947
0.57895
0.62842
0.67789
0.72737
0.77684
0.82632
0.87579
1.03177
1.11674
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K/P 32

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APPENDIX III

STOKES' LAW FOR PARTICLE SIZE DETERMINATION

A measurement of the size of the particles that was independent of the light transmission theory was necessary to provide a reference with which the results could be compared. Visual measurement with a microscope was found to be quite adequate for the experiments with graphite particles in a water medium. However, for smaller particles, particularly those whose physical chemistry would not permit them to be examined out of their own environment, another type of measurement was the application of Stokes' Law as follows.

If a small spherical particle of radius r is moving through a medium of viscosity η at a constant velocity v , the force that opposes this motion is given by Stokes' Law as

$$F = 6\pi\eta rv$$

for $Re \ll 1$. If the only other force acting on the particle is that of gravity, then the force tending to keep the particle in motion is given by

$$F = mg = \frac{4}{3}\pi r^3 (\rho - \rho')g$$

where ρ is the density of the particle in question and ρ' is the density of the suspending medium. Since these are the only two forces acting on the particle, they may be equated so that

$$\frac{4}{3}\pi r^3 (\rho - \rho')g = 6\pi\eta rv \quad \text{or,}$$
$$r = \sqrt{\frac{9\eta v}{2(\rho - \rho')g}}$$

Thus, by measuring the velocity at which the particles fall, the particle size may be determined.

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13. ABSTRACT		
<p>A light transmission technique was used to measure the mean size of a group of graphite particles suspended in water and the particle sizes in an aerosol of ammonium chloride in air. Passage of a single red laser beam through a known concentration of graphite particles in water made it possible to obtain a measure of their mean size. Laser beams of two different wavelengths were used for the measurement of a suspension of unknown particle size and concentration.</p> <p>Operating procedures were established for the apparatus, and calibration tests were performed in order to demonstrate the feasibility of the technique.</p>		

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